UNCLASSIFIED
CONFIDENTIAL



RM E56C16a

Copy
RM E56C16a

NACA RM E56C16a

NACA

# RESEARCH MEMORANDUM

## SELF-IGNITION TEMPERATURES AND FLASH POINTS OF SOME HIGH-ENERGY FUELS (U)

By Barbara Pusanski

Lewis Flight Propulsion Laboratory
Cleveland, Ohio

CANCELLED
Classification Changed
CHANGED TO UNCLASSIFIED
By authority of NASA Class. Chg #7 9/15/60
Date 10-14-64

CLASSIFIED DOCUMENT

This material contains information affecting the National Defense of the United States within the meaning of the espionage laws, Title 18, U.S.C., Secs. 793 and 794, the transmission or revelation of which in any manner to an unauthorized person is prohibited by law.

NATIONAL ADVISORY COMMITTEE
FOR AERONAUTICS
WASHINGTON
February 13, 1957

TECHNICAL LIBRARY
AIRESEARCH MANUFACTURING CO.
9851-9951 SEPULVEDA BLVD.
LOS ANGELES 45
CALIFORNIA

CONFIDENTIAL
UNCLASSIFIED

NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

RESEARCH MEMORANDUM

# SELF-IGNITION TEMPERATURES AND FLASH POINTS OF SOME HIGH-ENERGY FUELS

By Barbara Pusanski

## SUMMARY

The investigation of the ignition behavior of eight commercially prepared high-energy fuels containing boron, hydrogen, and carbon involved the determination of self-ignition temperatures by Setchkin's method and the determination of flash points by a closed-cup method using a small-scale closed-cup apparatus devised at the NACA Lewis laboratory.

The high-energy fuels, which were pure compounds rather than mixed fuels, showed a general trend toward lower flash point with lower boiling point, just as hydrocarbons do. However, they also showed a trend toward lower self-ignition temperature with lower boiling point, which is opposite to that of hydrocarbons.

## INTRODUCTION

There is current interest in compounds of boron, hydrogen, and carbon as potential high-energy fuels because of their high heat of combustion and their extreme reactivity in the combustion reaction. Such reactivity and ease of ignition are desirable properties for combustion in an engine, but they present problems in fuel handling. Therefore, a study of the ignition behavior of commercially prepared high-energy fuels was undertaken. Included in the study were the determination of self-ignition temperatures by Setchkin's method (ref. 1), and the determination of flash points by a closed-cup method using an apparatus devised at the Lewis laboratory.

The self-ignition temperature of a substance is the lowest temperature at which the substance will ignite in air or oxygen without the application of a source of ignition. Self-ignition temperatures of a single substance, as reported by different investigators, usually vary widely, depending on the type of procedure employed. A comprehensive survey of the methods used for the determination of self-ignition temperatures is presented by Mullins in reference 2, in which he has summarized the data from many of these methods and commented on the theory and significance of the methods. The self-ignition temperatures reported herein were determined by a single investigator in one apparatus under consistently similar experimental conditions.

The flash point is the lowest temperature of a liquid which will permit ignition of its vapor by an external source. As with self-ignition temperatures, there is considerable variation among values determined by different methods, and the flash points reported herein were obtained by a single investigator. A survey of flash point determination methods can be found in reference 3.

The self-ignition temperatures and/or flash points of the following high-energy fuels are reported herein: pure ethyl pentaborane (55Z4), two samples of pure ethyl decaborane (55Z1 and 55Z7), four samples of mixed fuels (55Z5, 55Z6, 55Z8, and 55Z9), and pure decaborane. Although dimethyl dipropyl silane is not a high-energy fuel, its self-ignition temperature is reported, since it is a fuel of high reactivity.

## MATERIALS

The high-energy fuels used in this investigation were commercially prepared. The dimethyl dipropyl silane was synthesized at the Lewis laboratory. The fuels are listed in table I. The four fuels described as mixed fuels contain diethyl decaborane and other polyalkylated derivatives of decaborane as impurities, but have ethyl decaborane as their principal constituent.

## APPARATUS AND PROCEDURE

Self-ignition temperature apparatus. - Self-ignition temperatures were determined in equipment (fig. 1) manufactured according to the design of an apparatus developed at the National Bureau of Standards (ref. 1). The ignition chamber is an electrically heated 1-liter spherical flask. Three independently controlled electric heating coils are regulated by variable transformers, and provide heat for the neck of the flask as well as the upper and lower spherical portions of the flask. The temperature inside the flask is indicated by a thermocouple whose junction is about $1\frac{1}{2}$ inches from the bottom of the flask.

The heaters were calibrated by using five temperatures in the range $65^{\circ}$ to $430^{\circ}$ C to produce a set of temperature-against-voltage curves. These curves indicate the three voltages that must be applied to the heaters to attain any desired uniform temperature within the apparatus in the range $65^{\circ}$ to $430^{\circ}$ C.

In order to determine the extent of temperature variation within the apparatus, the temperature was recorded at various depths inside the flask for each of the five temperatures used in the heater calibration. The temperature was found to be uniform in the spherical part of the flask.

Self-ignition temperatures were determined for three hydrocarbons, and the results compared with those obtained by Setchkin (ref. 1) for the same hydrocarbons under the same conditions. The comparison is shown in table II. The temperatures determined at this laboratory are slightly lower than those reported in reference 1.

Procedure for determining self-ignition temperatures. - Self-ignition temperatures were determined by preheating the apparatus to a temperature above the self-ignition temperature of the liquid being studied, then slowly lowering the temperature, and, at intervals of $1^{\circ}$ or $2^{\circ}$ C, injecting a charge of the combustible liquid into the flask from a hypodermic syringe and needle until a temperature was found below which ignition no longer occurred. The charge volume was usually 0.1 or 0.2 cubic centimeter. After each test the flask was flushed with an air stream in order to blow out the combustion products and provide a fresh atmosphere for the next test. In the case of high-energy fuels which leave solid deposits of combustion products on the walls of the flask, a clean flask was provided for each test temperature as the self-ignition temperature was approached. This was done in order to eliminate any effect which the solid coating might have on the self-ignition temperature.

Time-temperature records of the ignition of some of the high-energy fuels were obtained by connecting the thermocouple inside the flask to a photoelectric recorder.

Flash-point apparatus. - A miniature closed-cup flash-point tester was designed and fabricated at this laboratory so that flash points could be determined using small quantities of high-energy fuels. This apparatus incorporates the essential features of the standard Tag Closed-Cup Tester (ref. 4), but requires only $1\frac{1}{4}$ cubic centimeters of liquid to give the same vapor-to-liquid volume ratio as the 50 cubic centimeters needed for the standard Tag Tester. One cubic centimeter may be used as a more convenient volume. The nickel cup has a capacity of 3 cubic centimeters. An additional feature is a stirrer with two sets of impellers for mixing liquid and vapor; this was adapted from the Pensky-Martens Closed Tester (ref. 5) to assure mixing of the vapors and air in the space above the liquid. In figure 2 are a drawing of the apparatus and a diagram showing the operation of the flameholder. The stirrer rotates at 100 rpm. On the lid are a slide shutter and a gear arrangement, which simultaneously opens the shutter and depresses the tip of the flameholder into the opening above the cup.

To check the miniature apparatus against the standard Tag Closed Tester, six hydrocarbons were run in each. Table III gives the resulting flash-point data. Also cited in table III are some flash points obtained from reference 6. The results from the miniature tester are in fairly good agreement with those obtained from the standard Tag apparatus,

although most of them do not check well with the literature values. However, reference 6 does not explain the conditions or the apparatus used to determine these flash points, except to state that closed-cup methods were used.

A flash-point apparatus proposed by Reaction Motors, Inc. (RMI) was also used for determining some flash points. The apparatus was described in information obtained directly from Reaction Motors, Inc. It consists of a modified cup which is used in the standard Tag apparatus. The cylindrical cup has a depth of $1\frac{3}{32}$ inches, with a hemispherical well of radius 0.24 inch in the center of the bottom of the cup. The volume of the cup is 60 cubic centimeters. One cubic centimeter of liquid was used for all flash points determined in the RMI-proposed apparatus.

Procedure for determining flash points. - In determining a flash point, the procedure was similar to the one employed with the standard Tag Tester (ref. 4). The apparatus was in a liquid bath maintained at an appropriate temperature. For fuels that flashed above room temperature, a silicone oil bath was used. To obtain lower temperatures, freezing mixtures of glycol-water-ice or carbon tetrachloride-chloroform-dry ice were used. Because of the test flame on the lid of the apparatus, it was necessary to use these nonflammable materials in the surrounding liquid bath. The fuel to be tested was placed in the cup, and the lid was immediately fastened to the block. The temperature of the block was slowly raised (about $1^{\circ}$ C per min) by heating the bath. At intervals of $1^{\circ}$ C the test flame was introduced into the vapor space of the cup, then immediately removed. Stirring was discontinued while the test flame was applied. This procedure was repeated until a temperature was reached at which the vapor could be seen to flash when the flame was introduced. Testing in the dark made it easier to see the flash. For repeat determinations, fresh samples were used.

Handling of high-energy fuels. - Special precautions had to be taken in the handling and transfer of high-energy fuels because of their possible oxidation in air. Small volumes (1 or 2 cc) were transferred under a helium atmosphere to vials which were then closed with rubber serum stoppers. Samples were removed from these vials with a hypodermic syringe and needle by simply piercing the stopper with the needle and drawing the desired volume into the syringe. The stopper sealed itself on removal of the needle. It was necessary to avoid contact between the fuels and the rubber stoppers for reasons discussed in the following section.

## RESULTS AND DISCUSSION

Self-ignition temperatures. - The self-ignition temperatures of the high-energy fuels studied are shown in table IV. Ignitions of ethyl pentaborane (55Z4), HEF-3 (55Z8), and pure ethyl decaborane (55Z1 and

55Z7) occurred as violent explosions accompanied by brilliant green flashes and followed by green and orange flames. Ignitions of the Hi-Cal fuels (55Z5, 55Z6, and 55Z9) were milder. Ignitions of dimethyl dipropyl silane were violent above 265° C, but rather mild at lower temperatures. The two samples of pure ethyl decaborane, Z-197 and Z-272, had the same self-ignition temperature, and all four samples of mixed fuels had higher self-ignition temperatures than the pure ethyl decaborane. The three Hi-Cal fuels (55Z5, 55Z6, and 55Z9) showed successively higher self-ignition temperatures with each sample received. This result probably reflects modifications in preparation and purification of the fuel.

It was observed that pure ethyl decaborane samples which had been in vials for several days or weeks after their transfer gave longer ignition delays than freshly transferred samples. Furthermore, the ignition delays became longer as the time after transfer increased. In some cases, delays up to 10 or 12 minutes were noted. The samples of pure ethyl decaborane, colorless when first transferred, became yellow within a few days, and the color gradually darkened to a deep, clear amber.

In order to determine the cause of this increasing delay with time, three samples of Z-197 (55Z1), all transferred at the same time, were given different treatment over a period of 25 days. All three samples were clear and colorless on the day of transfer; the initial volume of each was 0.5 cubic centimeter. Sample A was undisturbed. The stopper in sample B was pierced with a hypodermic needle, but there was no contact between the sample and the stopper, and no liquid was withdrawn. The stopper in sample C was not punctured, but the vial was tipped over to bring the liquid in contact with the stopper for 10 seconds. The treatment described for samples B and C was performed 10 times during the 25-day period: on the day of transfer, and on the first, second, third, fourth, seventh, eighth, ninth, tenth, and fifteenth days after transfer. After 25 days, the samples were run in the Setchkin apparatus using a charge of 0.05 cubic centimeter at a temperature of 135° C. The results are shown in the following table:

| Sample (0.5 cc) | Treatment | Appearance after 25 days | Ignition delay |
|---|---|---|---|
| A | Undisturbed | Unchanged; volume unchanged | 28 sec |
| B | Contact with air | Cloudy white; volume unchanged | 15 sec |
| C | Contact with rubber stopper | Deep amber, clear; volume decreased to < 0.1 cc | 7 min 32 sec |

The long ignition delay, the color change, and the decrease in volume of sample C indicate that the cause of the observed aging effect in pure ethyl decaborane is a reaction between the sample and the rubber stopper.

Sample B apparently underwent slow oxidation or hydrolysis due to the introduction of small amounts of air or moisture at the time the stopper was pierced, but this had no effect on the ignition delay. The white cloudiness (presumably oxides of boron) in the liquid was considered to have been formed by contact with traces of oxygen or water.

Typical time-temperature records of the ignitions of the high-energy fuels in the Setchkin apparatus showed a single peak, which coincides with the time of ignition. Many of the time-temperature curves for pure ethyl decaborane were of this single-peak variety. However, some of them showed double or triple peaks. A few such curves for pure ethyl decaborane are reproduced in figure 3. In all the curves, the actual ignition or explosion coincides with the first peak. It should be noted that only the samples of pure ethyl decaborane exhibited multiple-peak time-temperature records. The four samples of mixed fuels produced ordinary single-peak curves. However, no explanation of the significance of these time-temperature records can be offered.

Flash points. - Flash points of some high-energy fuels, as determined in the miniature closed-cup tester, are given in table V. The high-energy fuels which are pure compounds (ethyl pentaborane, ethyl decaborane and decaborane) show a general trend toward lower flash points with lower boiling points; in this respect they resemble the hydrocarbons. No such trend is apparent in the case of the mixed fuels. The two samples of pure ethyl decaborane show a considerable difference in flash point, although they had the same self-ignition temperature.

The sample designated Z-197 had been stored at the Lewis laboratory for 7 months before the flash point (59° C) was determined, whereas the flash point (73° C) for Z-272 was determined within a month of the time the sample was received. The history of sample Z-244 (flash point 45° C) is not as well known, but it had come to the Lewis laboratory from Wright Air Development Center and had been here 2 months before the flash point was measured. These limited data indicate that changes occurred during storage of these fuels, which resulted in a change to a lower flash point.

The flash points of three of the high-energy fuels (Z-244, Z-197, and Z-272) were also determined in the RMI-proposed flash-point tester, using a 1-cubic-centimeter sample, and these data are included in table V. This apparatus did not distinguish differences in these samples as was noted above with the miniature closed-cup tester.

In order to test the performance of the miniature flash-point apparatus more thoroughly, the flash points of some hydrocarbon mixtures, prepared by adding small percentages of a low-boiling hydrocarbon (n-heptane or isopentane) to a higher-boiling hydrocarbon (n-dodecane), were determined in both the miniature apparatus and the standard Tag Closed-Cup Tester. The mixtures were also tested in the RMI-proposed flash-point apparatus, using 1 cubic centimeter. For convenience, 1 cubic centimeter had been used in the miniature apparatus for all previous flash-point determinations, and this volume was used for the hydrocarbon mixtures as well. However, these tests were repeated with a sample of 1.25 cubic centimeters, since this gives the miniature apparatus the same vapor-to-liquid volume ratio as that of the standard Tag apparatus. The resulting data, as shown in table VI, indicate that the miniature apparatus gives flash points for these hydrocarbon mixtures comparable to those obtained in the standard Tag apparatus, although the change in volume to $1\frac{1}{4}$ cubic centimeters introduced minor differences in flash points. However, two of the high-energy fuels, Z-272 and Z-244, when rerun in the miniature apparatus using a volume of $1\frac{1}{4}$ cubic centimeters, gave the same flash points as when 1 cubic centimeter was used.

The data in table VI from the hydrocarbon mixtures indicate the difficulty of determining reliable flash points for mixtures whose constituents vary widely in vapor pressure. Since the high-vapor-pressure constituents afford the greater hazard in the presence of an ignition source, it is important that the test procedure should be capable of detecting such constituents even when they are present in small quantities.

The mixtures of 1 and 2 percent isopentane in dodecane were selected for this part of the investigation to show the importance of the vapor-air volume in the sample cup above the liquid. With both of these mixtures it was calculated that a flammable mixture would not be produced in the RMI-proposed apparatus even by evaporation of all the isopentane alone from a 1-cubic-centimeter sample; whereas in the miniature closed-cup apparatus a flammable mixture should be formed by evaporation of the isopentane into the vapor space in the cup.

The experimentally determined flash points for these mixtures (table VI) agree with this expectation. The results of tests in the miniature cup did not agree exactly with those in the standard Tag apparatus, but they were consistent and reproducible. The results with these mixtures in the RMI-proposed apparatus varied widely and were not reproducible.

The results obtained for n-heptane - n-dodecane mixtures agreed satisfactorily among the three apparatus.

Therefore, it was concluded from both calculations and experiments that: (1) The vapor-air volume is critical for evaluating samples that

contain small amounts of components which have much higher vapor pressures than the rest of the mixture; and (2) the vapor-air volume probably will not affect the results obtained for mixtures in which the components have similar vapor pressures.

## CONCLUDING REMARKS

It has been noted that the high-energy fuels which are pure compounds have lower flash points with lower boiling points, and in this respect resemble the hydrocarbons. However, these high-energy fuels also tend to have lower self-ignition temperatures with lower boiling points, and this is opposite to the trend for hydrocarbons. This probably signifies that there is a product of thermal decomposition which ignites readily. Or perhaps it is simply a matter of the relative ignition energy levels of the two classes of compounds. In any case, this difference illustrates the fact that it is not safe to rely on a single test in judging the characteristics of a compound or series of compounds. A number of well-defined tests should be used.

## SUMMARY OF RESULTS

The self-ignition temperatures of seven boron-containing high-energy fuel samples were determined by Setchkin's method, and their flash points were determined in a miniature closed-cup apparatus. The following results were obtained:

1. Self-ignition temperatures for the high-energy fuels tested ranged from 92° C for ethyl pentaborane (55Z4) to 145° C for modified Hi-Cal-2 (55Z9).

2. Flash points ranged from -35° C for ethyl pentaborane (55Z4) to 73° C for pure ethyl decaborane Z-272 (55Z7).

3. The miniature closed-cup flash-point tester gave results which compare favorably with data obtained from the standard larger-capacity Tag Closed Tester in parallel tests where a number of pure hydrocarbons and hydrocarbon mixtures were used to compare the two apparatus.

4. The high-energy fuels which are pure compounds rather than mixed fuels were found to resemble the hydrocarbons in that lower-boiling fuels had lower flash points. However, the trend shown by self-ignition temperature data was opposite to that for hydrocarbons, since the lower-boiling high-energy fuels also had lower self-ignition temperatures, while lower-boiling hydrocarbons have higher self-ignition temperatures.

5. The flash point of ethyl decaborane samples (Z-272, Z-197, and Z-244) appeared to decrease as the storage time of the sample increased over a period of several months.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, March 19, 1956

## REFERENCES

1. Setchkin, Nicholas P.: Self-Ignition Temperatures of Combustible Liquids. Jour. Res. Nat. Bur. Standards, vol. 53, no. 1, July 1954, pp. 49-66.

2. Mullins, B. P.: Spontaneous Ignition of Liquid Fuels. AGARDograph No. 4, Butterworths Sci. Pub. (London), 1955.

3. Anon.: The Science of Petroleum. Vol. II. Oxford Univ. Press, 1938, pp. 1414-1416.

4. Anon.: Standard Method of Test for Flash Point by Tag Closed Tester. A.S.T.M. Designation D 56-52, A.S.T.M. Standards on Petroleum Products and Lubricants, Nov. 1952, pp. 1-6.

5. Anon.: Standard Method of Test for Flash Point by Means of the Pensky-Martens Closed Tester. A.S.T.M. Designation D 93-52, A.S.T.M. Standards on Petroleum Products and Lubricants, Nov. 1952, pp. 34-39.

6. Doss, M. P.: Physical Constants of the Principal Hydrocarbons. Fourth ed., The Texas Co., 1943

7. Schechter, William H., Jackson, C. B., and Adams, Roy M.: Boron Hydrides and Related Compounds. Second ed., Callery Chem. Co., May 1954.

8. Tannenbaum, Stanley, Kaye, Samuel, and Lewenz, George F.: Synthesis and Properties of Some Alkylsilanes. Jour. Am. Chem. Soc., vol. 75, no. 15, Aug. 5, 1953, pp. 3753-3757.

9. Voegtly, R. O., et al.: Preliminary Investigations of Methods of Preparing Alkyl Derivatives of Pentaborane-9 and Decaborane. Rep. No. CCC-1024-TR-80, Callery Chem. Co., Dec. 27, 1954. (Contract NOa(s) 52-1024-c.)

TABLE I. - FUELS INVESTIGATED

| Fuel | Prepared by | Commercial fuel name or number | NACA fuel number | Reference for fuel properties |
|---|---|---|---|---|
| Pure decaborane | Olin-Mathieson (purified by NACA) | ----- | ----- | 7 |
| Pure ethyl pentaborane | Callery | ----- | 55Z4 | No published data |
| Pure ethyl decaborane | Olin-Mathieson | Z-197 | 55Z1 | No published data |
| Pure ethyl decaborane | Olin-Mathieson | Z-272 | 55Z7 | No published data |
| Mixed fuel | Callery | Hi-Cal-2 | 55Z5 | No published data |
| Mixed fuel | Callery | Modified Hi-Cal-2 | 55Z6 | No published data |
| Mixed fuel | Olin-Mathieson | Z-244 or HEF-3 | 55Z8 | No published data |
| Dimethyl dipropyl silane | NACA | ---------- | ---- | 8 |

TABLE II. - COMPARISON OF SELF-IGNITION TEMPERATURE DATA FOR HYDROCARBONS

| Hydrocarbon | Self-ignition temperature in air, °C | |
|---|---|---|
| | Setchkin (ref. 1) | NACA data |
| n-Pentane | 258 | 253 |
| n-Nonane | 211 | 206 |
| n-Hexadecane | 202 | 199 |

TABLE III. - COMPARISON OF FLASH-POINT DATA FOR HYDROCARBONS

| Hydrocarbon | Flash point, °C (±1° C) | | |
|---|---|---|---|
| | Ref. 6 | Miniature tester (using 1 cc) | Standard Tag (using 50 cc) |
| n-Nonane | 31 | 34 | 33 |
| Isopropyl benzene | 39 | 33 | 30 |
| 1-Methyl-4-isopropyl benzene | 47 | 54 | 51 |
| p-Diethyl benzene | 59 | 59 | 58 |
| n-Dodecane | 74 | 80 | 78 |
| n-Hexadecane | 110 | 121 | 122 |

TABLE IV. - SELF-IGNITION TEMPERATURES OF HIGH-ENERGY FUELS

| Fuel | NACA fuel number | Charge, cc | Ignition delay, Min | Ignition delay, Sec | Temperature, °C | Self-ignition temperature, °C |
|---|---|---|---|---|---|---|
| Ethyl pentaborane | 55Z4 | 0.2 | | 6 | 92 | 92 |
| | | .2 | No ignition in 6 min | | 90 | |
| | | .2 | No ignition in 6 min | | 88 | |
| Ethyl pentaborane | 55Z4 | 0.1 | | 1 | 99 | 94 |
| | | .1 | | 3 | 94 | |
| | | .1 | No ignition in 6 min | | 92 | |
| | | .1 | No ignition in 6 min | | 86 | |
| Ethyl decaborane Z-197 (pure) | 55Z1 | 0.1 | | 8 | 132 | 123 |
| | | .1 | | 12 | 130 | |
| | | .1 | | 19 | 127 | |
| | | .1 | | 30 | 125 | |
| | | .1 | | 45 | 123 | |
| | | .1 | No ignition in 7 min | | 122 | |
| | | .1 | No ignition in 7 min | | 121 | |
| Ethyl decaborane Z-272 (pure) | 55Z7 | 0.1 | | 4 | 142 | 123 |
| | | .1 | | 22 | 133 | |
| | | .1 | 1 | 21 | 123 | |
| | | .1 | No ignition in 5 min | | 122 | |
| Z-244 or HEF-3 (Impure ethyl decaborane) | 55Z8 | 0.1 | | 25 | 133 | 130 |
| | | .1 | | 57 | 130 | |
| | | .1 | No ignition in 7 min | | 129 | |
| | | .1 | No ignition in 7 min | | 127 | |
| Hi-Cal-2 (Impure ethyl decaborane) | 55Z5 | 0.1 | | 1 | 132 | 125 |
| | | .1 | | 2.5 | 125 | |
| | | .1 | No ignition in 6 min | | 124 | |
| | | .1 | No ignition in 6 min | | 122 | |
| Hi-Cal-2 (Impure ethyl decaborane) | 55Z5 | 0.2 | | 3 | 126 | 126 |
| | | .2 | No ignition in 6 min | | 125 | |
| | | .2 | No ignition in 6 min | | 124 | |
| Modified Hi-Cal-2 (Impure ethyl decaborane) | 55Z6 | 0.1 | | 3 | 145 | 142 |
| | | .1 | | 3 | 143 | |
| | | .1 | | 5 | 142 | |
| | | .1 | No ignition in 5 min | | 141 | |
| | | .1 | No ignition in 5 min | | 140 | |
| Modified Hi-Cal-2 (Impure ethyl decaborane) | 55Z9 | 0.1 | | 9 | 146 | 145 |
| | | .1 | | 18 | 145 | |
| | | .1 | No ignition in 5 min | | 144 | |
| | | .1 | No ignition in 5 min | | 133 | |
| Dimethyl dipropyl silane | ----- | 0.2 | | 12 | 288 | 263 |
| | | .2 | | 16 | 283 | |
| | | .2 | | 34 | 274 | |
| | | .2 | | 50 | 271 | |
| | | .2 | 1 | 22 | 266 | |
| | | .2 | 2 | 58 | 264 | |
| | | .2 | 3 | 20 | 263 | |
| | | .2 | No ignition in 6 min | | 261 | |
| | | .2 | No ignition in 6 min | | 260 | |

TABLE V. - FLASH POINTS OF HIGH-ENERGY FUELS

| Fuel | NACA fuel number | Miniature apparatus flash point,[a] °C | RMI apparatus flash point,[d] °C | Boiling point, °C | Reference for boiling point |
|---|---|---|---|---|---|
| Pure compounds | | | | | |
| Ethyl pentaborane | 55Z4 | -35 to -25 | ---- | 104 | 9 |
| Decaborane[b] | ---- | 50±1 | ---- | 213 | 7 |
| Ethyl decaborane, Z-197 | 55Z1 | 59±1 | 70±1 | 283 | (c) |
| Ethyl decaborane, Z-272 | 55Z7 | 73±1 | 71±1 | 283 | (c) |
| Mixed fuels | | | | | |
| Hi-Cal-2 | 55Z5 | 17±1 | ---- | 236 | (e) |
| Modified Hi-Cal-2 | 55Z6 | 55±1 | ---- | 225 | (e) |
| Modified Hi-Cal-2 | 55Z9 | -17±3 | ---- | --- | - |
| Z-244 or HEF-3 | 55Z8 | 45±1 | 71±1 | 242 | (e) |

[a]All determinations were performed in the miniature flash-point apparatus using 1 cc of liquid.

[b]Decaborane was tested as a solid, since its flash point is below its melting point. Because this quantity filled the cup to about the same level as 1 cc of liquid, 0.5 g of crystals was used. (The melting point is 99.5° C, ref. 7.)

[c]This boiling point was determined on a different sample of pure ethyl decaborane (Z-199) by the Olin-Mathieson Chemical Corp.; the data were received in a private communication. The actual extrapolated boiling point obtained was 300° C; this was corrected for the capillary action of mercury on glass to give 283° C.

[d]For flash-point determinations in the RMI apparatus, 1 cc of liquid was used.

[e]Obtained at the Lewis laboratory.

TABLE VI. - COMPARISON OF FLASH-POINT DATA

FOR HYDROCARBON MIXTURES

| Composition of mixture, percent by volume | Flash point, °C (±1° C) | | | |
|---|---|---|---|---|
| | Standard Tag (50 cc) | Miniature apparatus (1.25 cc) | Miniature apparatus (1 cc) | RMI-proposed apparatus (1 cc) |
| 100% n-Dodecane | 78 | 80 | 80 | 78 |
| 1% Isopentane, 99% n-Dodecane | 7 | 15(±2) | 15(±2) | (a) |
| 2% Isopentane, 98% n-Dodecane | 6 | 14(±2) | 14(±2) | (b) |
| 1% n-Heptane, 99% n-Dodecane | 61 | 61 | 62 | 61 |
| 2% n-Heptane, 98% n-Dodecane | 53 | 53 | 55 | 53 |
| 3% n-Heptane, 97% n-Dodecane | 48 | 48 | 49 | 48 |
| 5% n-Heptane, 95% n-Dodecane | 40 | 40 | 46 | 40 |
| 10% n-Heptane, 90% n-Dodecane | 29 | 35 | 35 | 29 |

[a]Duplicate determinations could not be obtained. The results from five separate determinations were 29°, 49°, 56°, 75°, and 76° C.

[b]Duplicate determinations could not be obtained. The results from four separate determinations were 27°, 35°, 61°, and 70° C.

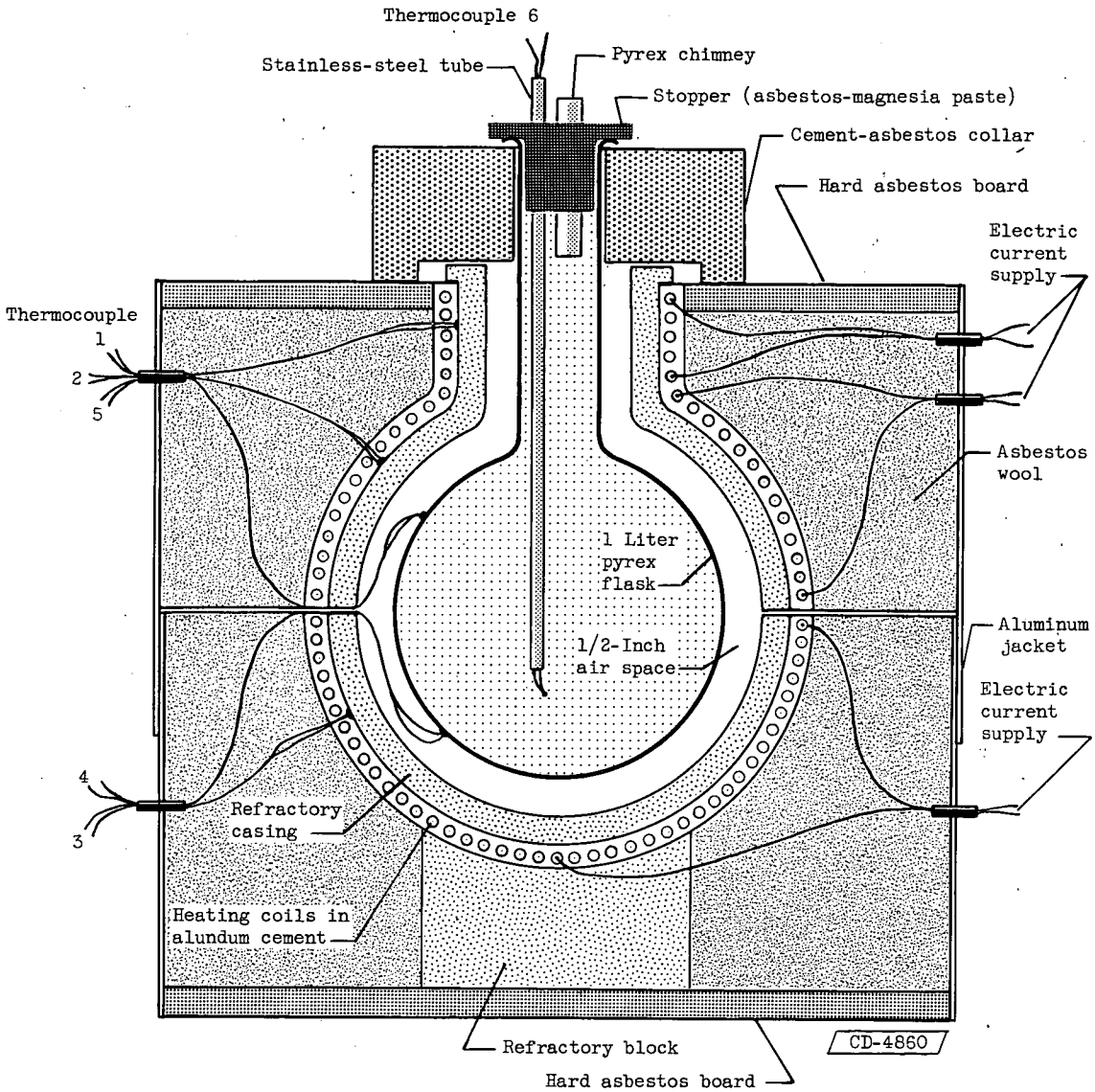


Figure 1. - Setchkin's self-ignition-temperature apparatus (ref. 1).

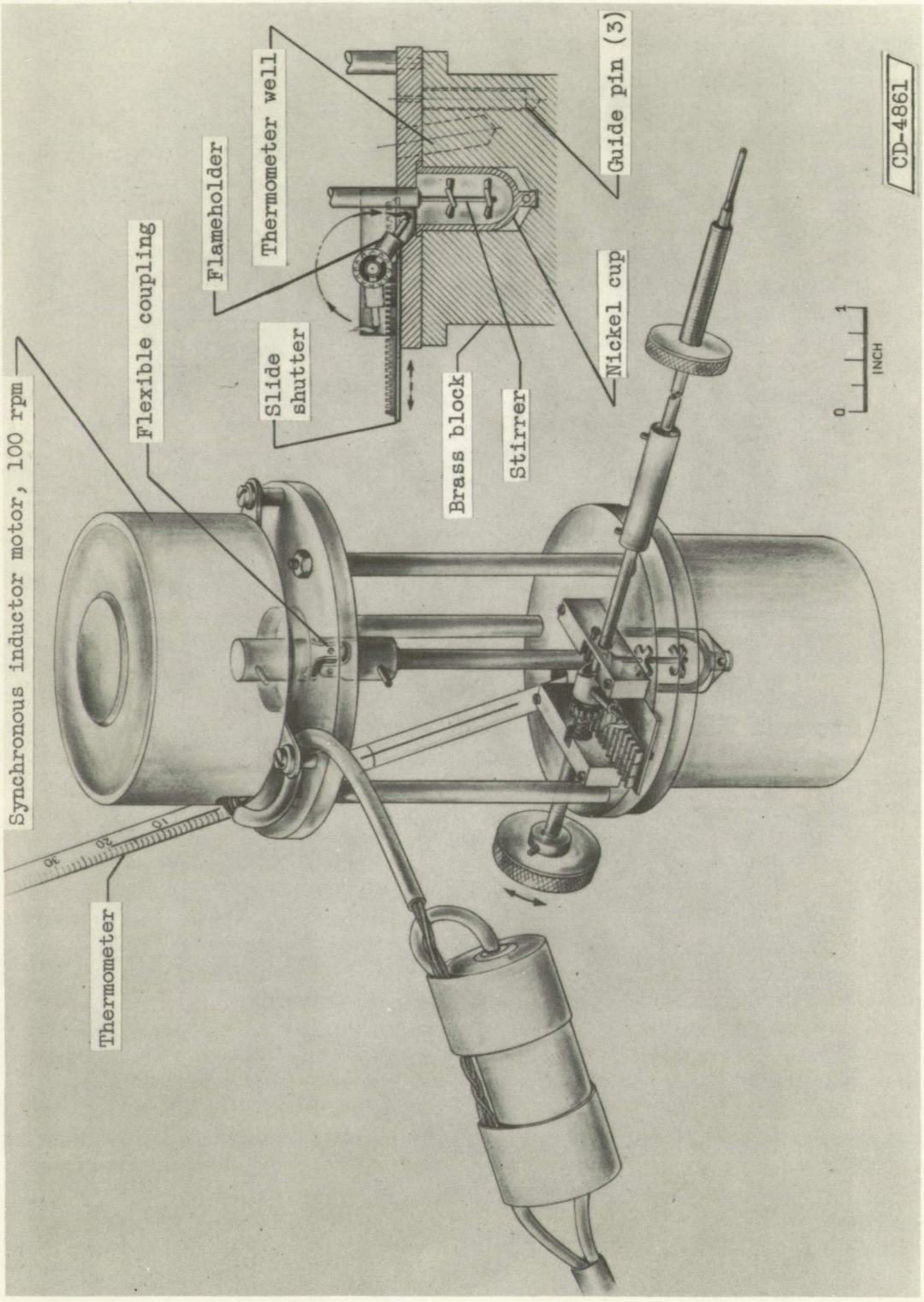


Figure 2. - Miniature flash-point apparatus.

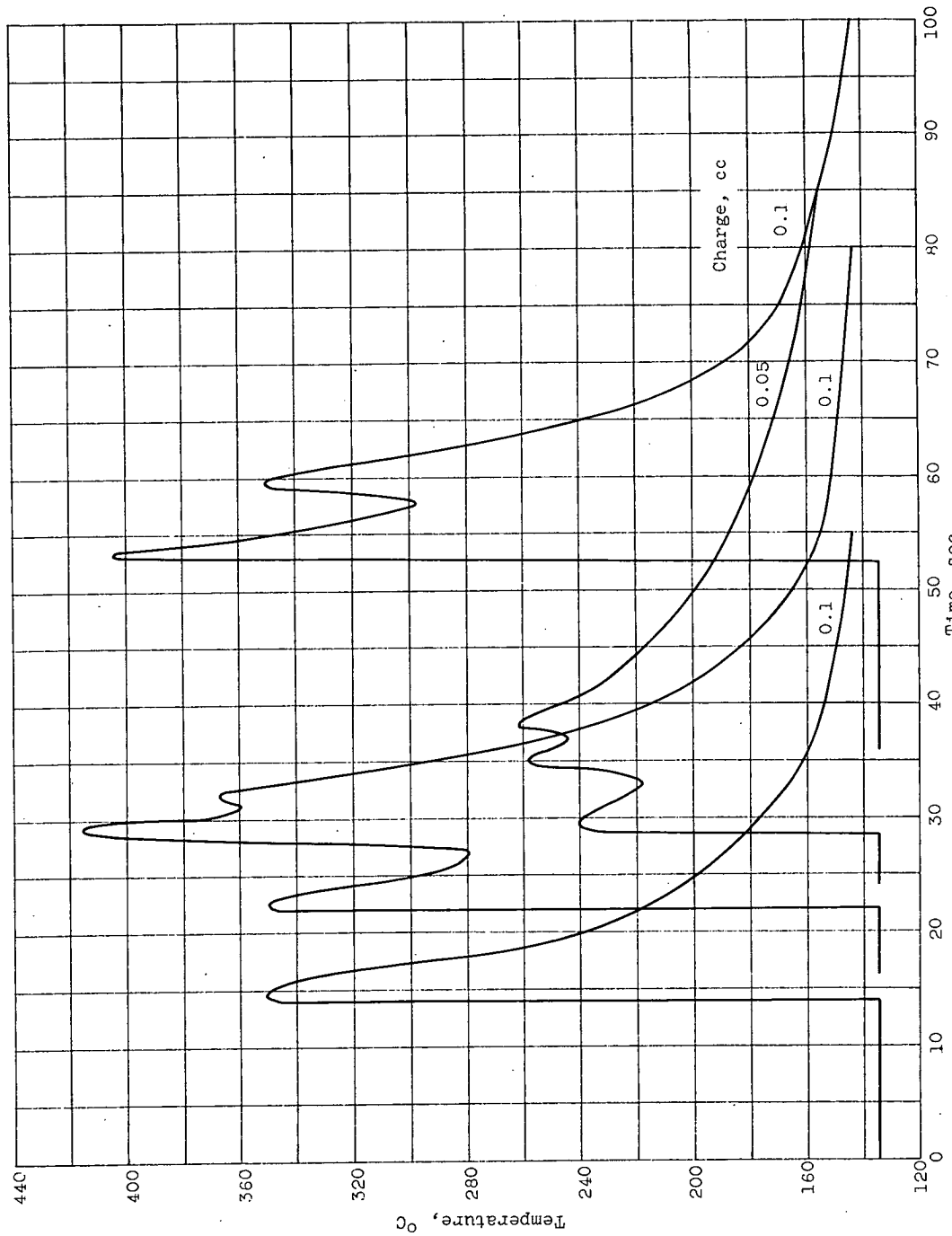


Figure 3. - Time-temperature curves for pure ethyl decaborane (55Z1) obtained in the Setchkin apparatus. All runs at 135° C.



NACA - Langley Field, Va.

CONFIDENTIAL
UNCLASSIFIED

CONFIDENTIAL
UNCLASSIFIED